

# Corrosion Resistance of API5L X52 Carbon Steel in Sulfide Polluted Environments

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## Abstract

**In the present work, the corrosion behavior of base and welded API5L X-52 carbon steel coupons in synthetic ground water solution NS-4 was studied. Subsequently, the morphology and composition of the corrosion product deposits were determined using scanning electron microscopy (SEM). Based on weight loss as well as Tafel's measurements, increasing the sulfide ions content above 0.4 mM, had an inhibition effect on the corrosion behavior of carbon steel. According to EDAX analyses, increasing of sulfide concentration from 0.4 to 4 mM Na<sub>2</sub>S, the Fe/S ratio decreased from 12.02 at 0.4 mM to 2.6 at 4 mM Na<sub>2</sub>S.**

## Keywords

**Carbon Steel, Sulfides, Weight Loss, Anodic Dissolution, Polarization**

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## 1. Introduction

The corrosion of carbon steel in sulfide-containing solutions has attracted widespread attention for many years due to its importance in several industrial processes such as oil and gas production and transport, petroleum refining, and petrochemical/chemical processing [1].

Some variables which affect the corrosion rate of steel in soil such as water, degree of aeration, pH, redox potential, resistivity, soluble ionic species, and microbiological activity. Concerning the environmental aspect, corrosion of steel structures in soil is a relevant problem due to soil and ground water contamination caused by failure of pipelines and other structures [2].

The presence of some pollutants, mainly sulfide, in the oil can affect the performance of mild steel in the petrochemical industry, as this phenomenon is responsible for costly economic and human loss. Sulfide pollution of ground water can occur from the industrial waste discharge, biological and bacteriological process from sea-

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weed, sulfide reduced bacteria, or industrial waste discharge [3] [4].

Other applications where sulfide species have produced corrosion include water production, electric power, and marine applications. In addition, the corrosion of steel in  $\text{CO}_2$ ,  $\text{NaCl}$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{NaHCO}_3$ , or  $\text{Na}_2\text{S}$  solutions constitutes the natural media often confronted in oil and gas prospecting and transportation areas [1].

The effect of sulfide on the corrosion behavior of carbon steel in natural waters is studied by many authors [5] [6]. In the present work, the electrolyte used is a synthetic solution to simulate the soil or underground water conditions (NS-4 solution). This environment considers groundwater conditions similar to the pipelines that are exposed to underground environments while used for transmission lines. The corrosion behavior of steel in soil environment is studied using the weight loss, open circuit measurements (OCP) and Tafel's extrapolation analysis. Subsequently, the morphology and composition of the corrosion product deposits are determined using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDX).

## 2. Experimental

### 2.1. Materials

Experiments were conducted using API5L X-52 carbon steel pipes, provided by GASCO company-Egypt, with outer diameter 168.275 mm and the thickness 6.9 mm. The chemical composition and mechanical characteristics of this steel are given in **Table 1** and **Table 2**. The hardness of the material was 155 HVN, and the microstructure was composed of pearlite and ferrite as shown in **Figure 1**. For corrosion measurements, rectangular base as well as weld-coupons with dimensions  $200 \times 40$  mm, were prepared and were subjected to grinding using Sic paper up to grid 1200 and polishing using alumina paste. In order to investigate the effect of plastic deformation, U-bend samples were also prepared with dimensions  $200 \times 40 \times 6.9$  mm. Three different diameters rolls of

**Table 1.** Chemical composition of API5LX-52 pipeline steel.

C	Si	P	S	Mn	Ni	Cr	Cu	Mo	V	Al
0.21	0.22	0.015	0.008	0.56	0.029	0.04	0.064	0.01	0.003	0.005

**Table 2.** Mechanical characteristics of API5LX-52 pipeline steel.

Specimen	W (mm)	Thickness (mm)	Yield Strength (Mpa)	UTS (Mpa)	Strain %
Weld	18.8	6.2	n.a	567	n.a
Base Metal	11.5	6.9	355	527	25



**Figure 1.** Microstructure of API5L X52 base metal (4'' pipeline material).

30 mm, 40 mm, and 50 mm were used in the universal testing machine to give three different strains and hence three different stresses (**Table 3**).

## 2.2. Electrolytes

The synthetic underground water (NS-4) electrolyte used was near-neutral pH environment (pH 8.4), whose chemical composition is given in **Table 4** after Parkins *et al.* [7]. The sulfide stock solution was prepared by dissolving ( $\text{Na}_2\text{S} \cdot 9 \text{H}_2\text{O}$ ) crystals in NS-4 solution. The effect of sulfide ions concentration on the corrosion behavior of carbon steel was studied in NS-4 solution containing 0.4, 2, 4, and 10 mM  $\text{Na}_2\text{S}$  with conductivity values 1456, 2230, 2680 and 5030  $\mu\text{S}$ , respectively.

## 2.3. Immersion Test

The corrosion performance of carbon steel coupons in synthetic underground water (NS-4), contaminated with different sulfide contents, was studied by measuring the weight loss as well as the open circuit potential (OCP) at time intervals of 7 days for 3 month. Samples were rinsed with distilled water and carefully dried before taking weight change measurements. The corresponding corrosion rates (CR) in mils per year (mpy), based on weight loss measurements, were calculated using the following equation [8]:

$$\text{CR} = 534 \frac{W}{DA t} \quad (1)$$

where W is the weight loss in gm (g), D is the density of steel  $\text{g}/\text{cm}^3$ , A is the total surface area ( $\text{cm}^2$ ) and t is the total exposure time in hours.

## 2.4. Potentiodynamic Test

The potentiodynamic test was conducted using a standard three electrodes cell. Each specimen was used as a working electrode with an exposure area of  $1 \text{ cm}^2$ . A saturated calomel electrode was used as a reference electrode and the counter electrode was a platinum electrode. The cell was connected to PARSATAT4000 potentiostat. The scanning range was set from  $-200 \text{ mV}$  to  $+200 \text{ mV}$  with respect to open circuit potential using a scanning rate  $0.5 \text{ mV}/\text{sec}$ . Versa Studio Software was used for drawing polarization curves, as well as Tafel's plot.

## 2.5. Surface Characterization

SEM (JEOL JXA-840A) was used to demonstrate the microstructure and surface morphology of the corrosion products. Local compositions were obtained using the energy dispersive spectroscopy (EDS) unit attached to the SEM.

**Table 3.** Roll Diameter vs. strain & stress obtained.

Roll Diameter	Strain	stress
30 mm	23%	158%SMYS
40 mm	17.25%	156%SMYS
50 mm	13.8%	152%SMYS

**Table 4.** Chemical composition of NS-4 synthetic ground water solution [8].

Chemicals	g/l
$\text{NaHCO}_3$	0.483
KCl	0.122
$\text{CaCl}_2$	0.093
$\text{MgSO}_4$	0.131

### 3. Results

#### 3.1. Open Circuit Potential (OCP)

Figures 2-4 summarize the potential measurements (OCP) for both base and weld metal specimens after immersion for 12 weeks in both working conditions (stressed and unstressed samples) as a function of sulfide ions content. It is concluded that the OCP values for stressed samples were more negative than the unstressed ones. The OCP values ranged from  $-680$  mV up to  $-730$  mV. There was a slight change in potential values by increasing the sulfide content, where the OCP values were in the range of  $\sim -680$  to  $-700$  mV. The most negative OCP was in case of NS-4 solution containing  $0.4$  mM  $\text{Na}_2\text{S}$ , as shown in Figure 4.

#### 3.2. Weight Loss Measurements

Figure 5 illustrates a summary of corrosion rate (mpy) based on weight loss measurements for base as well as weld metal coupons after immersion for 3 month, as a function of sulfide content. For base metal coupons, the highest corrosion rate was obtained in NS-4 +  $0.4$  mM  $\text{Na}_2\text{S}$  ( $0.09$  mpy). Increasing the sulfide ions content above  $0.4$  mM, had an inhibition effect on the corrosion behavior of carbon steel. The lowest corrosion rate ( $0.06$  mpy) was obtained in NS-4 +  $4$  mM  $\text{Na}_2\text{S}$ . Similarly, for weld metal coupons, the highest corrosion rate ( $0.18$  mpy) was obtained in NS-4 +  $0.4$  mM  $\text{Na}_2\text{S}$ . Weld metal coupons showed higher corrosion rate than its correspondents of base metal immersed in the same solutions.

#### 3.3. Potentiodynamic Test

The electrochemical parameters calculated using the Tafel plots (Figure 6) confirmed that the addition of sulfide increases the corrosion current,  $i_{\text{corr}}$  and consequently the corrosion rates (mpy). The anodic branches present a uniform dissolution for examined specimens in all solutions.

Corrosion data obtained from Tafel's plot are summarized in Table 5. From this table, the extent of increase in  $i_{\text{corr}}$  and corrosion rates (mpy) is found to be a function of the concentration of  $\text{Na}_2\text{S}$ . The addition of  $\text{Na}_2\text{S}$  has an inhibition effect of the corrosion behavior of carbon steel samples. This trend is in agreement with test result based on weight loss measurements. Also from Table 5, and in agreement with the OPC measurement, the most negative corrosion potential ( $-768$  mV) was obtained in NS-4 solution containing  $4$  mM  $\text{Na}_2\text{S}$ .

Corrosion rate (mpy) calculated from the Tafel's plots are presented in Figure 7. According to Figure 7, the weld metal specimen had higher corrosion rate than base metal one for both working conditions (stressed and

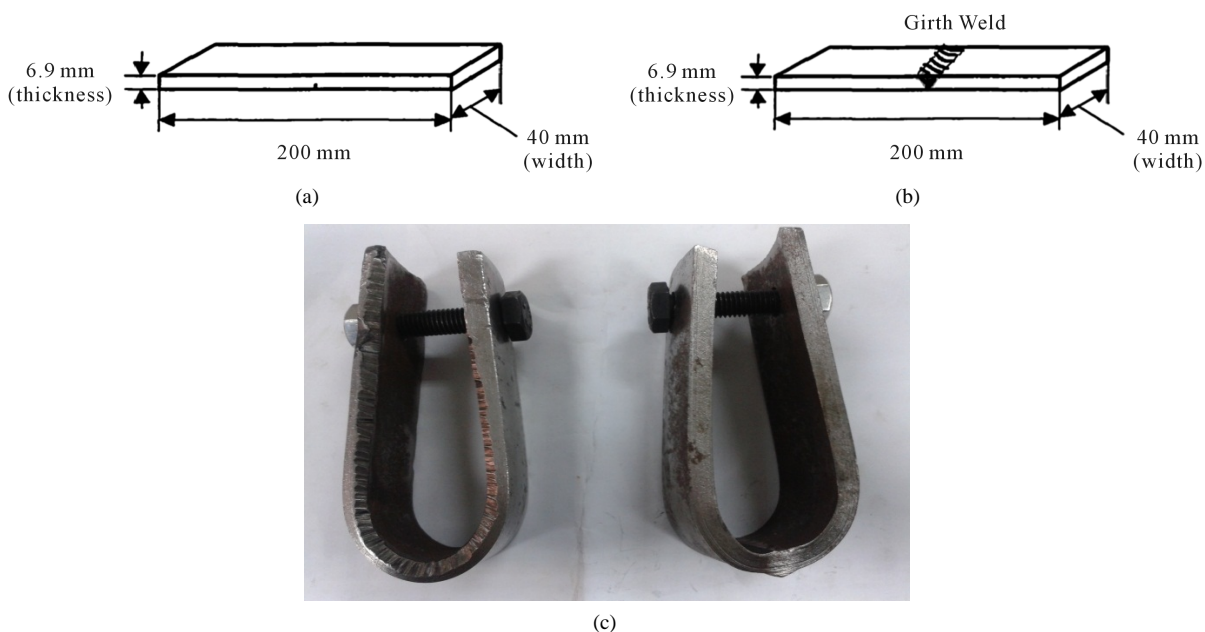
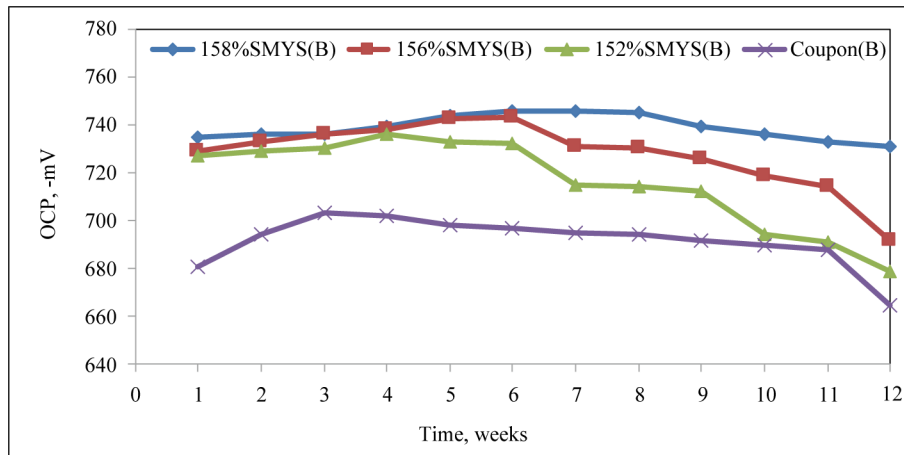
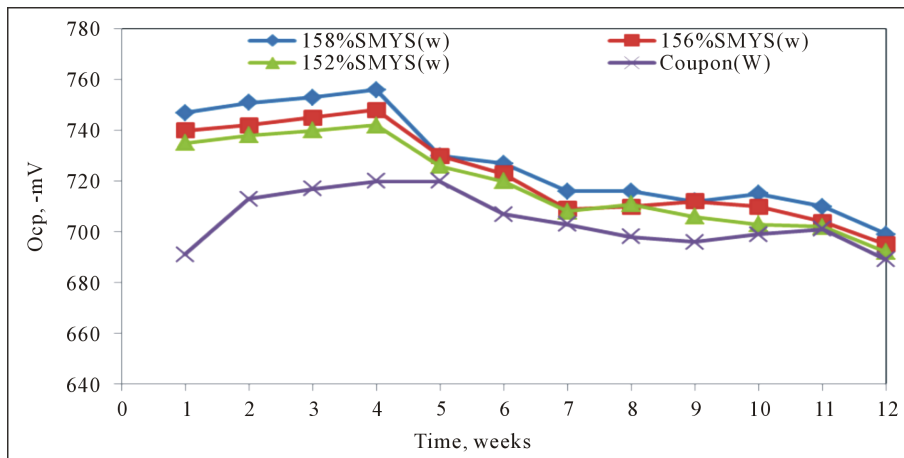


Figure 2. Test Specimens. (a) Base-metal specimen; (b) Weld Specimen; (c) U-bend stressed specimens.

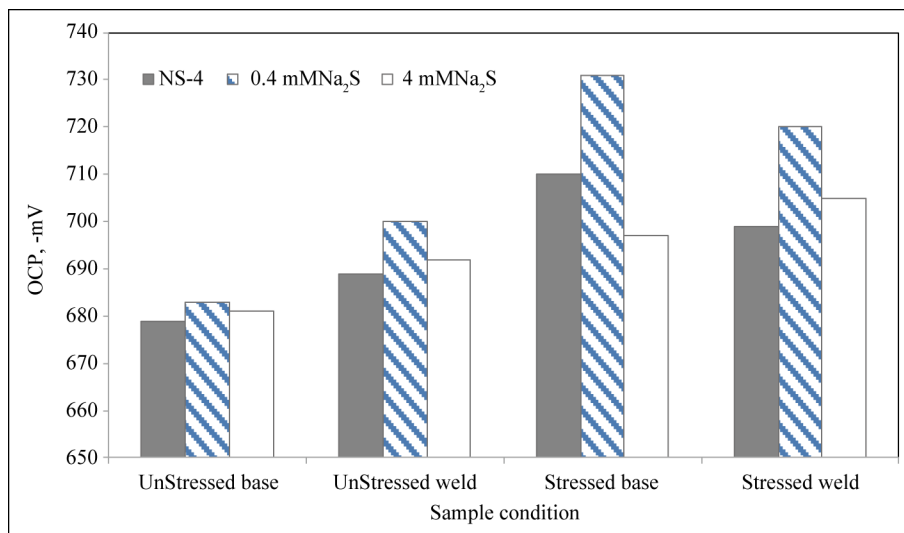


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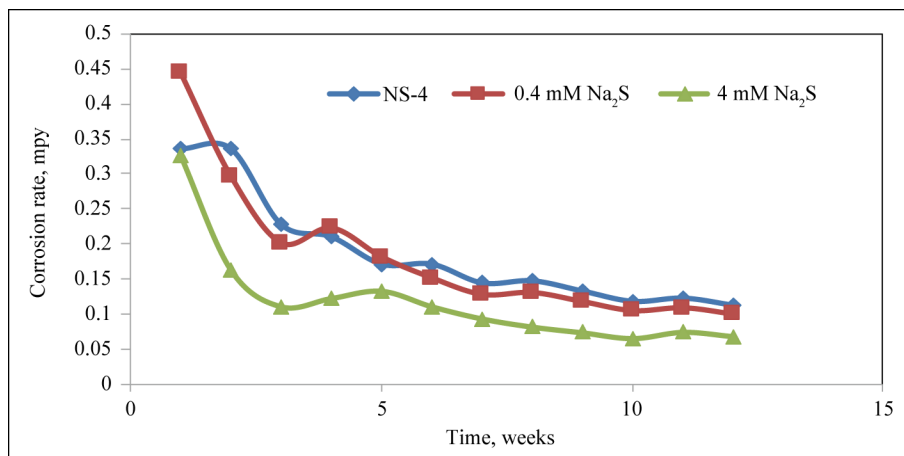


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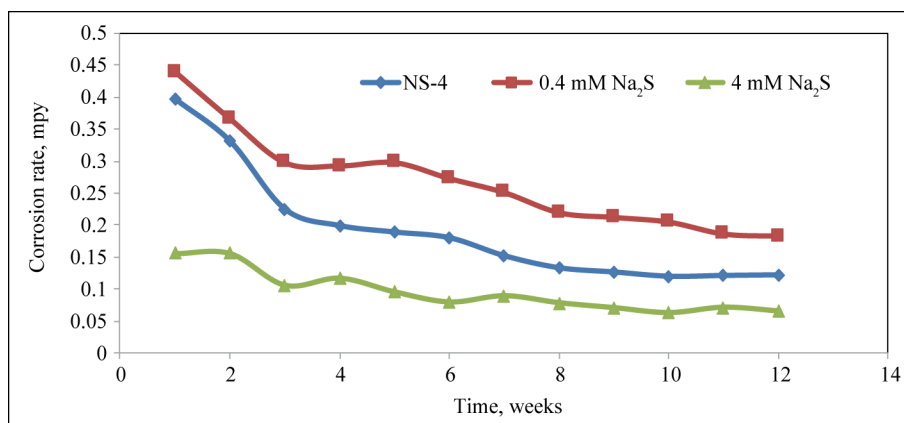
**Figure 3.** Effect of plastic deformation applied on OCP measurements in NS-4 solution. (a) Base-metal; (b) Welded-metal.



**Figure 4.** Open circuit potential of specimens for different sulfide-contaminated environments and different samples preparation conditions after immersion for 12 weeks.

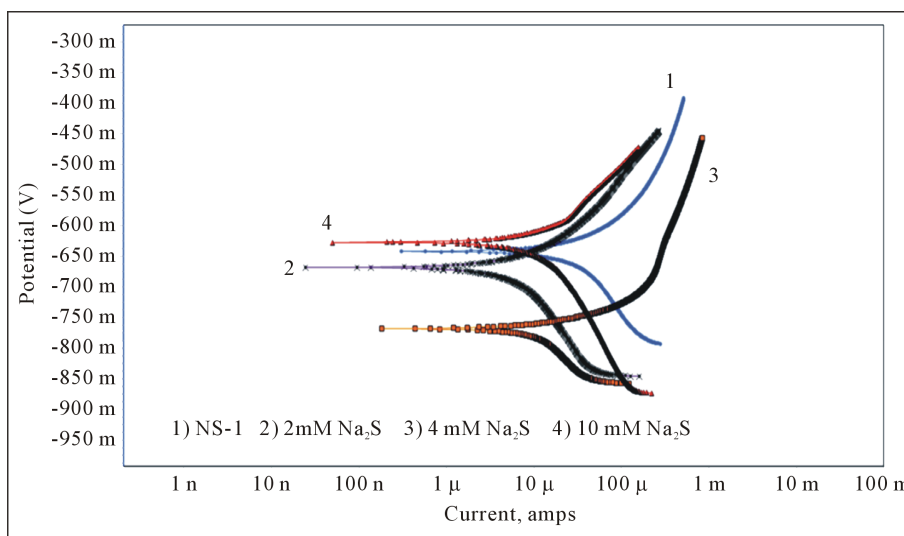


(a)

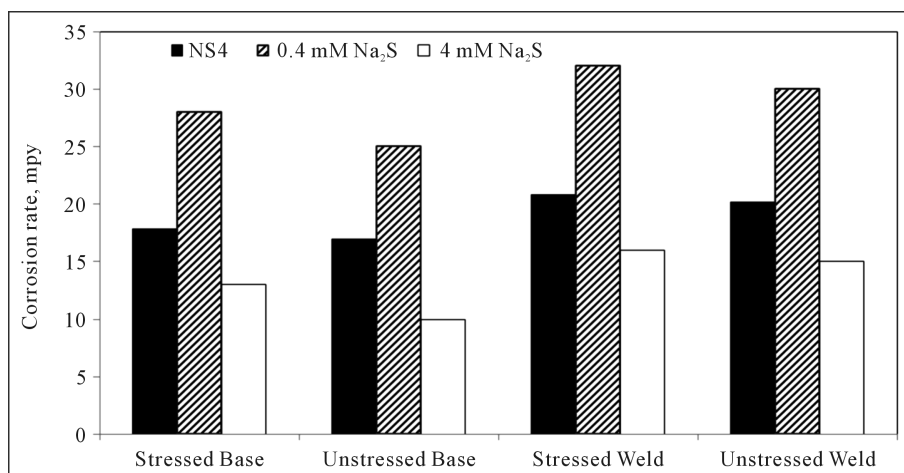


(b)

**Figure 5.** Corrosion rates (mpy) of welded-coupons after immersion in different sulfide-contaminated environments for 12 weeks-based on weight loss measurements. (a) Base metal; (b) Welded metal.



**Figure 6.** Polarization curves of weld coupons measured in sulfide contaminated environments.



**Figure 7.** Corrosion rates of carbon steel specimens as a function of sulfide content as well as stresses applied.

**Table 5.** Corrosion data of API5LX-52 welded coupons immersed in different sulfide polluted electrolytes.

Condition	$I_{corr}$ ( $A/\mu\text{cm}^2$ )	Corrosion rate (mils/year)	$E_{corr}$ (mV)	$\beta_a$ mv/decade	$\beta_c$ mv/decade
NS4	21.2	20.2	-642	130	160
NS4 + 0.4 mNa <sub>2</sub> S	31	30	-720	162	170
NS4 + 2 mNa <sub>2</sub> S	15.34	14.74	-667	151.5	200
NS4 + 4 mNa <sub>2</sub> S	15.5	15	-768	37.4	162.8
NS4 + 10 mNa <sub>2</sub> S	18.8	18.1	-627	180	175

unstressed). In case of stressed welded samples, the corrosion rates were 18, 32, and 15 mpy for NS-4 solutions with 0, 0.4, and 4 mM Na<sub>2</sub>S, respectively.

### 3.4. SEM Surface Morphology and EDAX Analysis

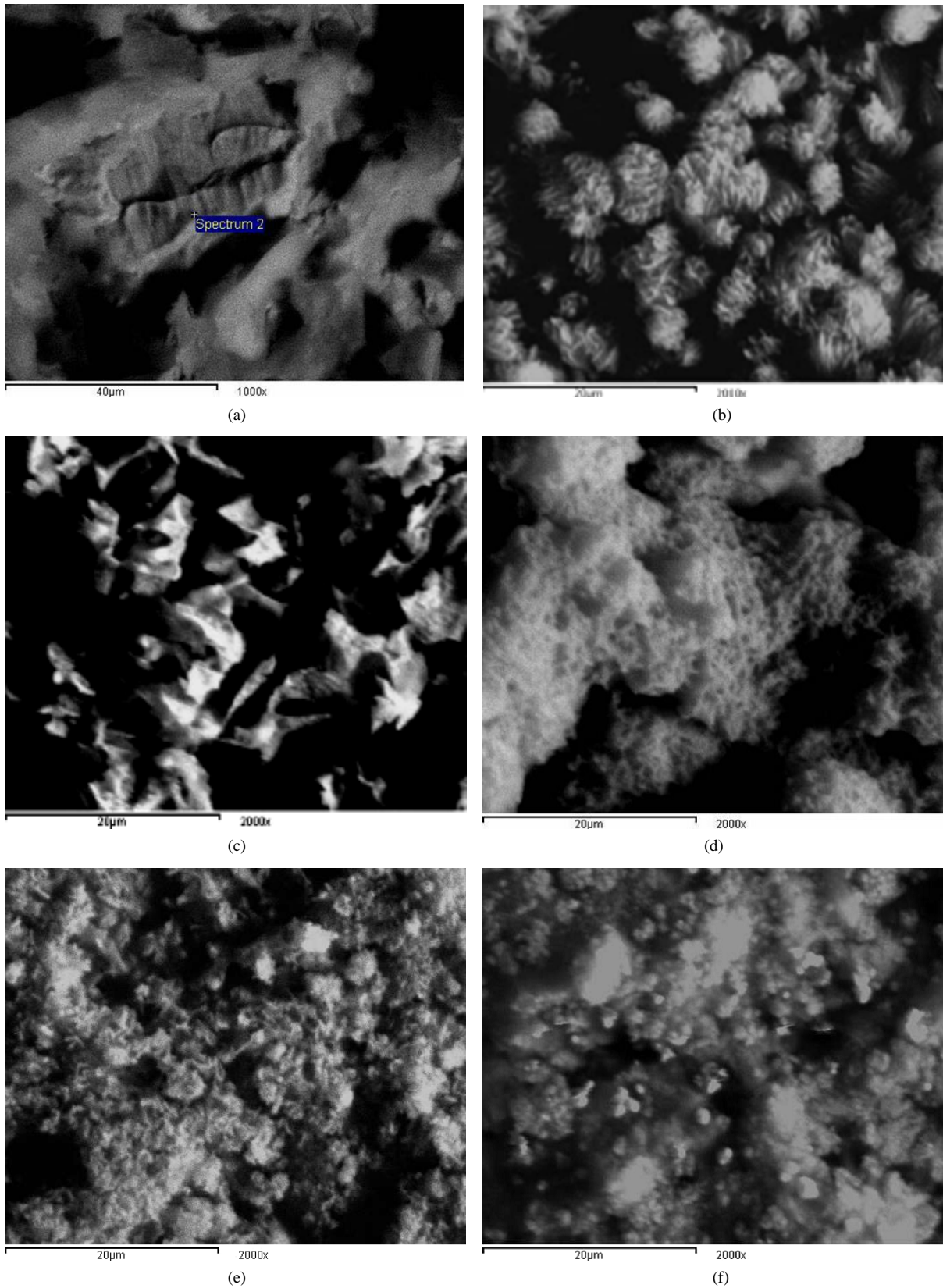
The SEM images of base as well as weld metal coupons after immersion for three month in different sulfide polluted environments are illustrated in **Figure 8**. The SEM micrograph of base metal coupon which was immersed in NS4 solution, is characterized by dense layer (**Figure 8(a)**). From **Table 6**, EDAX analyses showed a high content of iron (88.14%) and the presence of oxygen (6.05%), carbon (4.58%), and manganese (%1.23). On the other hand, the SEM micrograph of weld metal coupon (**Figure 8(b)**) is characterized by flowery shaped layer formed on the metal surface immersed in the same above environment.

**Figure 8(c)** shows the SEM micrograph of weld metal coupon which was immersed in NS-4 + 0.4 mM Na<sub>2</sub>S after 3 months. The SEM image shows a thin porous layer, and consequently non protective layer. EDAX analysis of this layer showed a lower content of iron (50.1%) and the presence of higher content of oxygen (32.7%), carbon (9.08%), magnesium (3.81%) and sulfur (4.17%), by increasing the sulfide content.

**Figure 8(d)** shows the SEM micrograph of the surface of the weld metal coupon which was immersed in NS-4 + 2 mM Na<sub>2</sub>S for 3 months. The SEM image shows a dense spongy layer formed on the metal surface.

**Figure 8(e)** shows the SEM micrograph weld metal coupon which was immersed in NS-4 + 4 mM Na<sub>2</sub>S for 3 months. The SEM image shows a dense layer with some bright particles formed on the metal surface. EDAX analysis (**Table 6**) indicated a lower iron content (41.47%) and the presence of of oxygen (27.9%), carbon (9.05%), magnesium (4.54%) and higher sulfur content (15.71%), in case of 4 mM Na<sub>2</sub>S.

**Figure 8(f)** shows the SEM micrograph of the surface of the weld metal coupon which was immersed in NS-4 + 10 mM Na<sub>2</sub>S for 3 months. The SEM image shows a dense protective layer with many bright particles formed on the metal surface.



**Figure 8.** SEM of weld-metal coupons after immersion in sulfide contaminated environments for 3 month.



**Table 6.** EDAX analysis of API5LX-52 coupons taken at the weld zones after 3 month of immersion.

Condition	% C	% O	% Mn	% Mg	% S	% Si	% Fe
NS-4	4.58	6.05	1.23	0.0	0.0	0.0	88.14
NS-4 + 0.4mM Na <sub>2</sub> S	9.08	32.79	0.0	3.81	4.17	0.0	50.15
NS-4 + 4 mM Na <sub>2</sub> S	9.05	27.90	0.0	4.54	15.71	0.0	41.47

#### 4. Discussion

According to this work, and from potentiodynamic polarization test, it was noticed that stressed specimens showed higher corrosion rate than unstressed coupons in both cases of base and weld metal specimens. This is in agreement with other study showing that the corrosion current increased significantly and monotonically for samples with increasing plastic deformation [9]. The value of applied strain was based on the following formula;

$$\varepsilon = t/2R \text{ when } t < R \quad (2)$$

where  $\varepsilon$  is the applied strain,  $t$  is the specimen thickness and  $R$  is the radius of the bend [10].

When steels are plastically deformed, some fraction of the deformation energy (~5%), is retained internally. The major portion of this stored energy is as strain energy associated with dislocations. The increase in the amount of strain energy would allow easier dissolution on specific sites since this energy is released on dissolution. This causes an increase in the corrosion rate with an increase in the amount of cold work [11].

It was also recognized from potentiodynamic polarization test, and in agreement with weight loss test, that weld metal specimen suffered higher corrosion rate than base metal ones. This can be arisen from the fact that weld-metal composition (which is normally optimized for mechanical properties) tend to be slightly anodic to the parent steel in certain environments. Therefore, the weld metal corrodes at a higher rate than the base metal [12] [13].

The deterioration of metal due to contact with hydrogen sulfide (H<sub>2</sub>S) and moisture is called sour corrosion which is the most damaging to drill pipe. Although H<sub>2</sub>S is not corrosive by itself, it becomes a severely corrosive agent in the presence of water, leading to pipeline embrittlement. Hydrogen sulfide when dissolved in water is a weak acid, and therefore, it is a source of hydrogen ions and becomes corrosive. The corrosion products are iron sulfides (FeS<sub>x</sub>) and hydrogen. Iron sulfide forms a scale that at low temperature can act as a barrier to slow corrosion. The forms of sour corrosion are uniform, pitting, and stepwise cracking. The general equation of sour corrosion can be expressed as follows [14]:



There is a possibility for formation of H<sub>2</sub>S in the presence of some sulfide salts, according to the following equations [15] [16];



Another possible mechanism for iron dissolution in aqueous electrolytes containing H<sub>2</sub>S is based on the formation of a tetragonal ferrous Sulfide (mackinawite) FeS as follows;

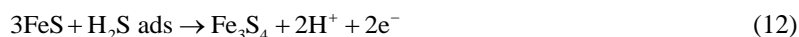


The FeS layer of steel is not stable, it is removed from the steel surface in an acidic environment, forming

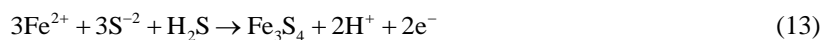
again H<sub>2</sub>S, enhancing corrosion:



There is a possibility that the surface of the steel will be covered by Fe<sub>3</sub>S<sub>4</sub> protective layer that blocks the carbon steel from further corrosion. This layer could be doped with other corrosion products:



Subsequently



This is in agreement with the study mentioned in the report from ICPTT [16], and explains the reduction in corrosion rate measured by weight loss as well as Tafel's extrapolation technique, by increasing the sulfide ions content.

Increasing the sulfide ions content above 0.4 mM, had an inhibition effect on the corrosion behavior of carbon steel. For base metal coupons, the highest corrosion rate was obtained in NS-4 + 0.4 mM Na<sub>2</sub>S (31 mpy). The lowest corrosion rate was obtained by conducting potentiodynamic test in NS4 + 4 mM Na<sub>2</sub>S (~15 mpy).

The weight loss test of coupons immersed in NS4 + 4 mM Na<sub>2</sub>S agreed with these results obtained from potentiodynamic test, which showed a low corrosion rate at high concentration of Na<sub>2</sub>S. The reason for this low corrosion rate may be because of the use of sodium sulfide as the source of H<sub>2</sub>S. At higher pH sodium sulfide may not produce H<sub>2</sub>S gas and the dominant species could be S<sup>2-</sup>, and the absence of H<sup>+</sup> ions decreases the corrosion rate. Also the reaction product, formed at higher pH is metal sulfides, which when precipitates on the surface of the metal gives protection to the metal as these metal precipitates have extremely low solubility. This is in agreement with a study performed by M. Koteeswaran [17] on CO<sub>2</sub> and H<sub>2</sub>S corrosion in oil pipe lines.

In addition, some results demonstrated that the presence of hydrogen sulfide can inhibit the corrosion of iron under certain special conditions due to the formation of a ferrous sulfide—pyrite protective film [18]-[20].

Recently, Sherar *et al.* [20] investigated the effect of inorganic sulfide on carbon steel corrosion in a solution containing chloride, bicarbonate and sulfate (pH 8.9) by following the evaluation of corrosion potential (E<sub>corr</sub>) and periodically measuring the polarization resistance (R<sub>p</sub>) over an exposure period of a few months. When freshly-polished carbon steel was exposed directly to sulfide a low corrosion rate was observed. However, when sulfide was added to pre-corroded steel, the corrosion rate tripled [21]. These observations were consistent with the results reported by Newman *et al.* [21], who hypothesized that a pre-corroded surface prevents FeS passivation.

The SEM image of the weld coupon immersed in NS-4 + 0.4 mM Na<sub>2</sub>S showed a thin porous layer which is not protective formed on the steel's surface, and this is in support of high corrosion rate in this solution. The SEM image of the weld coupon at high concentrations of Na<sub>2</sub>S showed a compact passive corrosion product film formed on the steel surface. The EDAX analysis showed that in case of NS-4 electrolyte, corrosion product formed on the metal surface was composed of; 4.58% C, 6.05% O, 1.23% Mn, 88.14% Fe. The protective product formed on the metal surface in case of 4 mM Na<sub>2</sub>S is composed of; 9.08% C, 32.79% O, 3.81% Mg, 4.17% S, 50.15% Fe. It was also noticed that with the increase of sulfide concentration from 0.4 to 4 mM, the Fe/S ratio decreased from 12.02 at 0.4 mM, to 2.63 at 4 mM Na<sub>2</sub>S. This shows that increasing the sulfide concentration leads to the formation of sulfur-rich iron sulfide [5].

## 5. Conclusions

- 1) Increasing the sulfide ions content above 0.4 mM Na<sub>2</sub>S, had an inhibition effect on the corrosion behavior of carbon steel, due to formation of protective film.
- 2) There was a slight change in potential values by increasing the sulfide content.
- 3) The protective product formed on the metal surface in case of NS-4 + 4 mM Na<sub>2</sub>S was composed of; 9.08% C, 32.79% O, 3.81% Mg, 4.17% S, 50.15% Fe.
- 4) Increasing of sulfide concentration from 0.4 to 4 mM, the Fe/S ratio decreased from 12.02 at 0.4 mM to 2.6 at 4 mM. This showed that increasing the sulfide concentration led to the formation of sulfur-rich iron sulfide.
- 5) The open circuit potential values for stressed steels were more negative than the unstressed samples.
- 6) Weldments of carbon steel showed more tendency for corrosion than base steel.

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